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Steel in Liquid Methane  
and in Liquid Natural Gas**

**Donald W. Wisander**

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## Friction and Wear of Selected Metals and Alloys in Sliding Contact With AISI 440C Stainless Steel in Liquid Methane and in Liquid Natural Gas

Donald W. Wisander  
*Lewis Research Center*  
*Cleveland, Ohio*



National Aeronautics  
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FRICTION AND WEAR OF SELECTED METALS AND ALLOYS IN SLIDING  
CONTACT WITH AISI 440C STAINLESS STEEL IN LIQUID  
METHANE AND IN LIQUID NATURAL GAS

by Donald W. Wisander

Lewis Research Center

SUMMARY

Various metals (aluminum, titanium, beryllium, nickel, iron, copper, and several alloys of copper) were run in sliding contact with 440C in liquid methane and liquid natural gas. There was no noticeable difference in the friction or wear when the same combination was run in liquid methane or in liquid natural gas.

All of the elemental metals except copper showed high wear and severe galling. Most of the wear tests produced ribbon-type wear debris which attached itself to the trailing edge of the rider wear scar. Copper had long thin ribbons; the copper-tin and copper-tin-lead had almost insignificant ribbons. The other metals had short, thick, curled ribbons of debris.

Friction coefficients observed varied from 0.2 to 1.0, the lowest being for the copper, copper-tin, and copper-tin-lead. The wear rate of copper was about two orders of magnitude lower than the other metals run (except for the copper-tin and copper-tin-lead alloys which were an order of magnitude less than the pure copper).

Examination of the photomicrograph of the wear scars revealed severe cold working and refinement of the crystal structure. The subsurface has a very fine crystal structure under which is a thin layer of heavily flowed material. This type structure generally existed for all metals run in this program.

The wear debris (trailing the wear scar) of the copper specimens had the characteristic laminar structure predicted by the recrystallization theory of wear.

INTRODUCTION

An economical form in which to store or to transport methane or liquid natural gas (LNG) is in the liquid state. Construction of storage facilities for liquid natural gas

(93 percent methane) and liquid methane (for peak shaving, base-load and for emergencies) continues to increase (refs. 1 to 5).

In addition to normal industrial uses, LNG is now used for fueling truck fleets and is being considered for automobiles (refs. 6 and 7) and advanced aircraft (refs. 8 and 9). In both LNG manufacture and distribution the liquid must be transferred by various pumps that have bearings and seals. For example, the automotive engine pumps to use LNG have mechanical parts that are in sliding or rolling contact such as bearings and/or seals. These components require materials with minimum wear. Low wear to these parts means not only longer component life but reduced contamination of the cryogen with wear debris. Wear debris contamination can accelerate the wear and erosion of other mechanical components of the end use product.

Results reported in reference 10 indicate that many metals in sliding contact in LNG show high friction, wear and severe galling and therefore are not lubricated by the LNG.

Experiments reported herein were conducted to determine which metals would give the best friction and wear in sliding contact with AISI 440C. These metals would then be alloyed in an attempt to further improve their lubricating properties. 440C was included as one of the sliding materials because it is a commonly used bearing alloy for use in cryogenic applications. The other metals used in this program included aluminum, titanium, beryllium, nickel, iron, copper, and several copper alloys.

These experiments were conducted using a hemisphere (4.76 mm in radius) sliding against the flat surface of a rotating disk (63.5 mm in diameter by 12.7 mm in thickness) operating submerged in the cryogen. Data presented is typical of two or three runs.

#### APPARATUS AND PROCEDURE

The apparatus used in the friction and wear studies is shown in figure 1. The basic elements consisted of a hemispherically tipped 4.76-millimeter-radius rider specimen held in sliding contact with the lower flat surface of a 63.5-millimeter-diameter rotating disk. The experiments were conducted with specimens completely submerged in liquid methane or liquid natural gas (LNG). The drive shaft supporting the disk specimen was driven by a hydraulic motor through a 6:1 speed increaser and provided a sliding velocity of 12.4 meters per second (4200 rpm) for the data reported herein. Two sets of helium-purged contact seals (not shown in fig. 1) were used to prevent air leakage in and cryogenic fluid leakage out around the drive shaft.

The cryogenic fluid was transferred to the test chamber through a closed system. The storage vessel was pressurized to transfer the liquid and to maintain the liquid

level in the test chamber.

The test chamber was cleaned with 90 percent ethyl alcohol prior to each run. After the cleaning and installation of specimens, the test chamber was closed, purged for 15 minutes with helium gas, and then filled with the liquid natural gas. After the test chamber was full and the liquid boiling stabilized, the rider specimen was loaded against the rotating disk to a load of 1 kilogram. The wear track diameter was about 56 millimeters. The duration of a standard test was 1/2 hour.

The frictional force and the load were measured by strain gage dynamometer rings. A two-pen recording potentiometer was used as a strain indicator. The wear of the rider specimen was determined by measuring the wear-scar diameter and calculating wear volume. The rider specimen displacement was measured by a linearly variable differential transformer and continuously recorded so that wear could be calculated at any specific time.

The surfaces of the metal disk specimens were prepared as follows: (1) finished-ground and lapped to  $5 \times 10^{-2}$  micrometer root mean square, (2) scrubbed with moist levigated alumina, (3) washed in tap water, and (4) washed in distilled water. The metal rider specimens were ground to  $10^{-1}$  micrometer root mean square and then cleaned by the same procedure as described for the disks (steps 2 to 4).

## MATERIALS

The Cu-2 wt. % Be and Cu-8 wt. % tin-22 wt. % lead are commercially manufactured alloys, the remaining alloys were prepared at NASA. NASA alloys were melted in argon and cast into a water-cooled copper mold. No heat treatment was used. Table I shows hardness values of the materials used in these experiments.

Both liquid methane and LNG were used for these experiments. The composition of the LNG is shown in table I.

## RESULTS AND DISCUSSION

The metals selected to be run against 440C were chosen either because they were useful aircraft materials (Al, Ti, Be) or were bearing alloy constituents (Ni, Fe in 440C; Cu, Ag in bearing bronzes). All elemental metals were 99.9 percent pure LNG. These metals were run in sliding contact with a 440C disk ( $R_c$  58).

Many of the experiments were performed in both liquid methane and in LNG to determine differences in friction and wear. Differences were so minor that, to avoid confusion, only methane is referenced. This observation is important when one considers the cost of liquid methane is about 10 times that of LNG.

## Friction and Wear of Elemental Metals

All of the elemental metals run against AISI 440C stainless steel showed high wear (fig. 2) and experienced severe galling and surface roughening (fig. 3) except copper and silver.

Attached to the trailing edge of the wear scar were streamers of wear debris. These streamers were generally weakly attached to the rider and easily lost due to handling. The 440C, titanium, nickel, and iron transferred significantly to the disk specimen. The other metals produced only a faint transfer film.

Silver and copper had wear rates that were two orders of magnitude less than the other elemental metals. They also had the lowest and smoothest friction coefficient.

Friction coefficients for the elemental metals are shown in figure 4. Nickel showed the highest and roughest friction coefficient, varying from 0.5 to 1.0.

Most interesting friction and wear was displayed by the copper. Friction coefficient for copper against 440C started at 0.3, after about 10 minutes the friction coefficient rapidly decreased to 0.16. This experiment was repeated with 10 different sets of copper 440C couples. The initial friction coefficient varied from 0.26 to 0.32 and the time for the reduction varied from 4 to 10 minutes. In all cases the friction decreased to about one-half of the initial value, and the friction trace was much smoother after the transition. (Explanation to follow.)

Wear calculation based on the recorded axial displacement of the rider showed that simultaneously with the friction drop was an accompanying wear rate decrease. Several of the runs were interrupted at various times before and after the friction transition. Wear scars before the transition were very rough, while after the transition the wear scars were smooth and highly polished. Wear tracks on the disks showed a faint copper-colored transfer film before and after the transition.

Calculations based on measurements of the rider wear from the trace (and also rider wear measurements from tests that were interrupted) revealed that the wear rate had decreased by a factor of 100 after the decrease in friction occurred. A representative graph of the friction coefficient and of the wear versus time is shown in figure 5.

Metallurgical examination of the rider (in the vicinity of the sliding zone) revealed that extensive recrystallization of the copper occurred (to a depth of 10  $\mu\text{m}$ ). This soft thin layer was the reason for the reduction in wear rate and friction coefficient (see photos in fig. 6). Further information on recrystallization theory can be obtained in metallurgy section and from reference 11.

It should also be noted that the copper riders had wear scars with trailing streamers of wear debris attached to trailing edge of wear scar. These streamers continue to "grow" only up to the time when the transition occurs. Photomicrographs of the edge of the streamers showed a layered structure with a recrystallized fine-grain

structure appearing on one surface of the streamer (fig. 6).

### Friction and Wear of Alloys

Copper (one of the lowest wearing metals) was alloyed with Be, Si, Al, and Al-Si (2 to 4.7 wt. %) to determine if hardening the copper would improve the wear resistance. The wear was not reduced (fig. 7) by the addition of these elements. Also the friction coefficients (fig. 8) were significantly higher, varying from 0.6 to 1.0; Cu-2 wt. % Be showed the highest friction coefficient.

Since copper is one of the most commonly used bearing metals, it was also run alloyed with tin and lead (commonly used alloying elements in bearing bronzes). The addition of 17 wt. % Sn and 8 wt. % Sn-22 wt. % Pb significantly reduced the wear rate of the copper (fig. 7). Friction coefficient (fig. 8) was not significantly affected. Of all the copper alloys, only the tin-copper showed the friction (and wear) transition revealed in the copper runs except to a lesser degree. Copper, copper-Sn and Cu-Pb all showed bright and smooth wear scars.

These results indicate that using the proper composition alloys of copper-tin or copper-lead are self-lubricating and can be effectively used in liquid methane sliding applications without the need for solid lubricants such as  $\text{MoS}_2$ .

### Metallurgy of Metal Specimens

A fine structure is seen near the subsurface of all materials examined. Earlier transmission electron microscopy studies performed on copper after sliding in liquid methane (ref. 12) indicated that the structure near the subsurface consisted of five cells about 0.5 to 1.0 micrometers in diameter. The interior of the cells were nearly void of dislocations, with a close-spaced dislocation array seen in some of the cell walls. This structure observed in copper was interpreted as resulting from a very rapid, high volume density rate of formation of recrystallization nuclei. The high volume density of nuclei was considered to arise with the aid of frictional heating, from the intensely cold worked material which was observed beneath the five structured layers. Coalescence of the nuclei could occur almost immediately, with very little growth taking place prior to the virtual completion of this rapid, local process described as recrystallization in reference 12.

It is likely that the fine structure observed near the subsurface of the materials shown in figure 9 resulted from a process very similar to the one described in reference 12. Variations in the process are a function of the temperature gradient near the

subsurface for the particular metal tested, as well as the basic physical and mechanical properties of the metal.

Hardness (table II) does not appear to be simply related to the recrystallization nor related to the friction and wear of the sliding couples run in these experiments.

#### SUMMARY OF RESULTS

Aluminum, titanium, beryllium, nickel, iron, copper, and several copper alloys were run in sliding contact with AISI 440C in liquid methane (and liquid natural gas) at 12.4 meters per second and 1 kilogram load. Results revealed the following:

1. Copper alloys with 4 percent lead or with 17 percent tin showed friction similar to unalloyed copper but had wear rates an order of magnitude lower.
2. Copper alloyed with beryllium, aluminum, or silicon (2 to 5 wt. %) shows greater friction and wear than pure copper.
3. Copper and silver showed friction coefficients similar to the other elemental metals but the wear rates were about two orders of magnitude lower. Friction of copper decreased to half of the initial value after 6 to 20 minutes, wear rate decreased by two orders of magnitude after the run-in period (6 to 20 min). This large change in wear rate is believed due to the recrystallization of the subsurface producing a soft layer possibly acting as a lubricant film.
4. Aluminum, titanium, beryllium, nickel, and iron showed high wear rates and friction coefficients from 0.3 to 0.5.

Lewis Research Center,

National Aeronautics and Space Administration,

Cleveland, Ohio, October 12, 1977,

505-16.

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TABLE I - COMPOSITION OF LIQUID  
NATURAL GAS USED FOR  
THESE STUDIES

[Source. NASA Mass Spectrometer.]

Hydrocarbon	Percent
CH <sub>4</sub>	93.2
C <sub>2</sub> H <sub>6</sub>	4.7
C <sub>3</sub> H <sub>8</sub>	.2
N <sub>2</sub>	1.8
C <sub>4</sub> H <sub>10</sub>	.08
C <sub>5</sub> H <sub>12</sub>	.08
O <sub>2</sub>	.02
Ar	
CO <sub>2</sub>	
H <sub>2</sub>	↓

TABLE II - ROOM TEMPERATURE  
HARDNESS OF METALS USED  
FOR THESE STUDIES

Elemental metals	Hardness
Nickel	R <sub>B</sub> 88
Titanium	R <sub>B</sub> 95
Beryllium	R <sub>B</sub> 77
Copper	R <sub>F</sub> 40
Silver	Vickers 33
Aluminum	R <sub>B</sub> 10
Iron	R <sub>B</sub> 45
Alloys	Hardness
Cu-2 wt. % Be	R <sub>C</sub> 40
Cu-4.7 wt. % Si	R <sub>B</sub> 67
Cu-4.5 wt. % al	R <sub>B</sub> 42
Cu-4.6 wt. % Al-2.4 wt. % Si	R <sub>B</sub> 43
Cu-17 wt. % Sn	R <sub>B</sub> 44
Cu-8 wt. % Sn-22 wt. % Pb	Brinnel 50
AIAI 440C stainless steel	R <sub>C</sub> 58

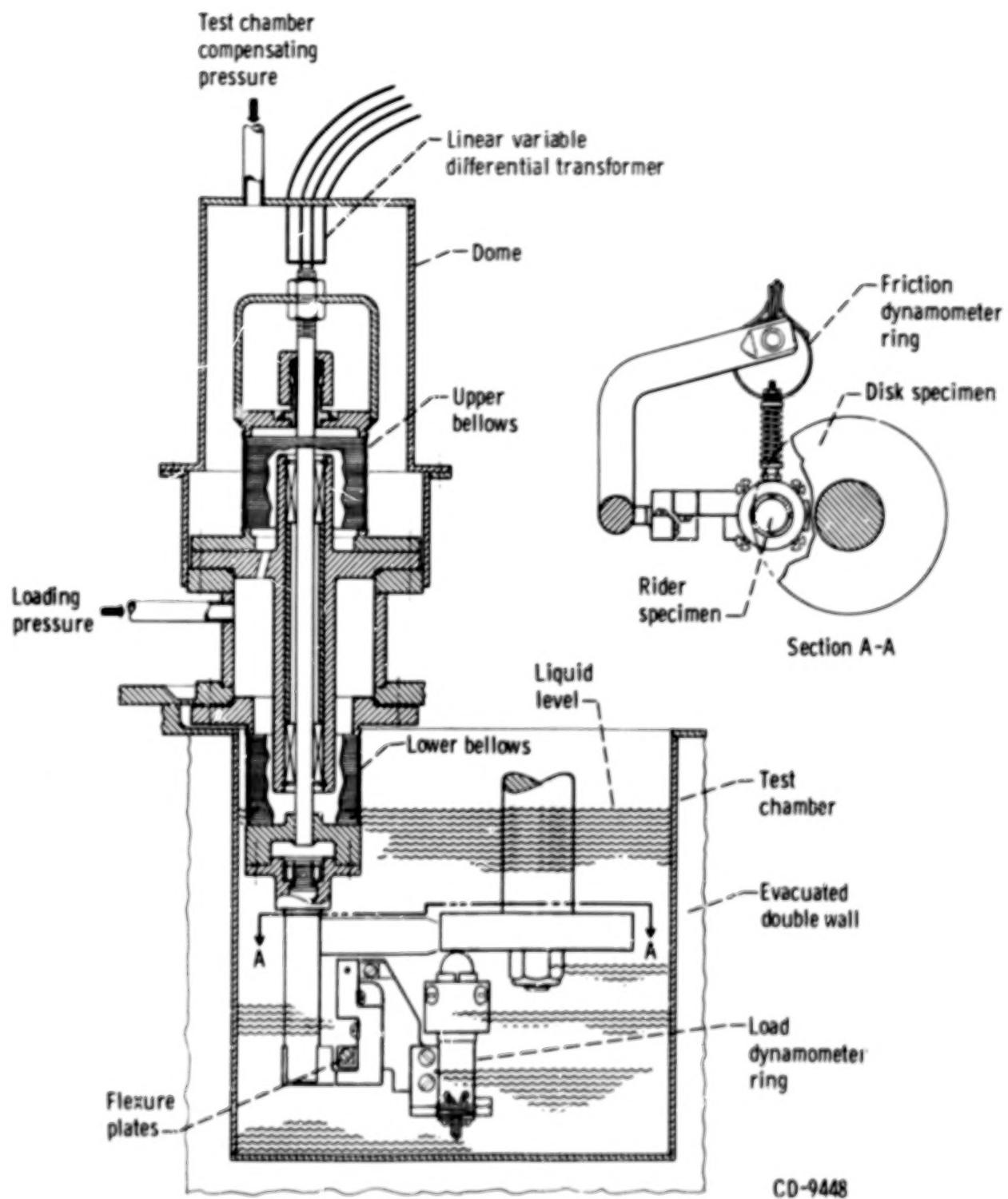


Figure 1. - Cryogenic fuel friction apparatus with specimen loading system.

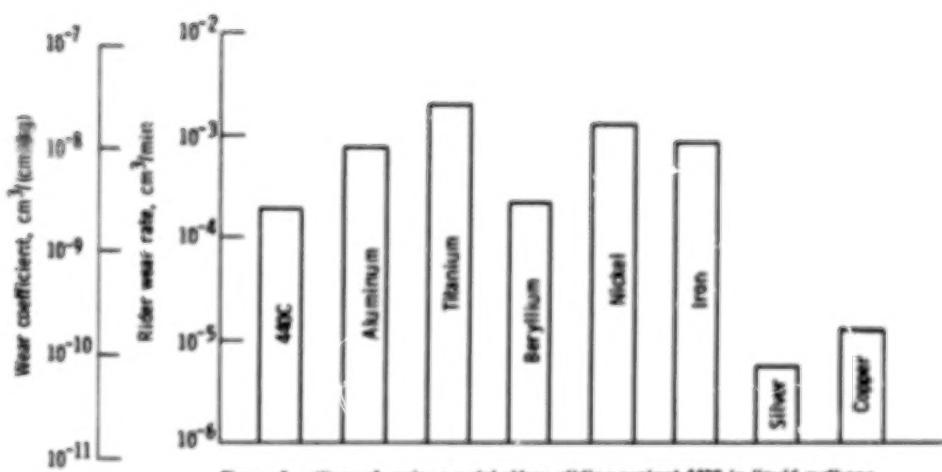


Figure 2. - Wear of various metal riders sliding against 440C in liquid methane.  
Load, 1 kilogram; sliding velocity, 12.4 meters per second (4200 rpm); duration 30 minutes.

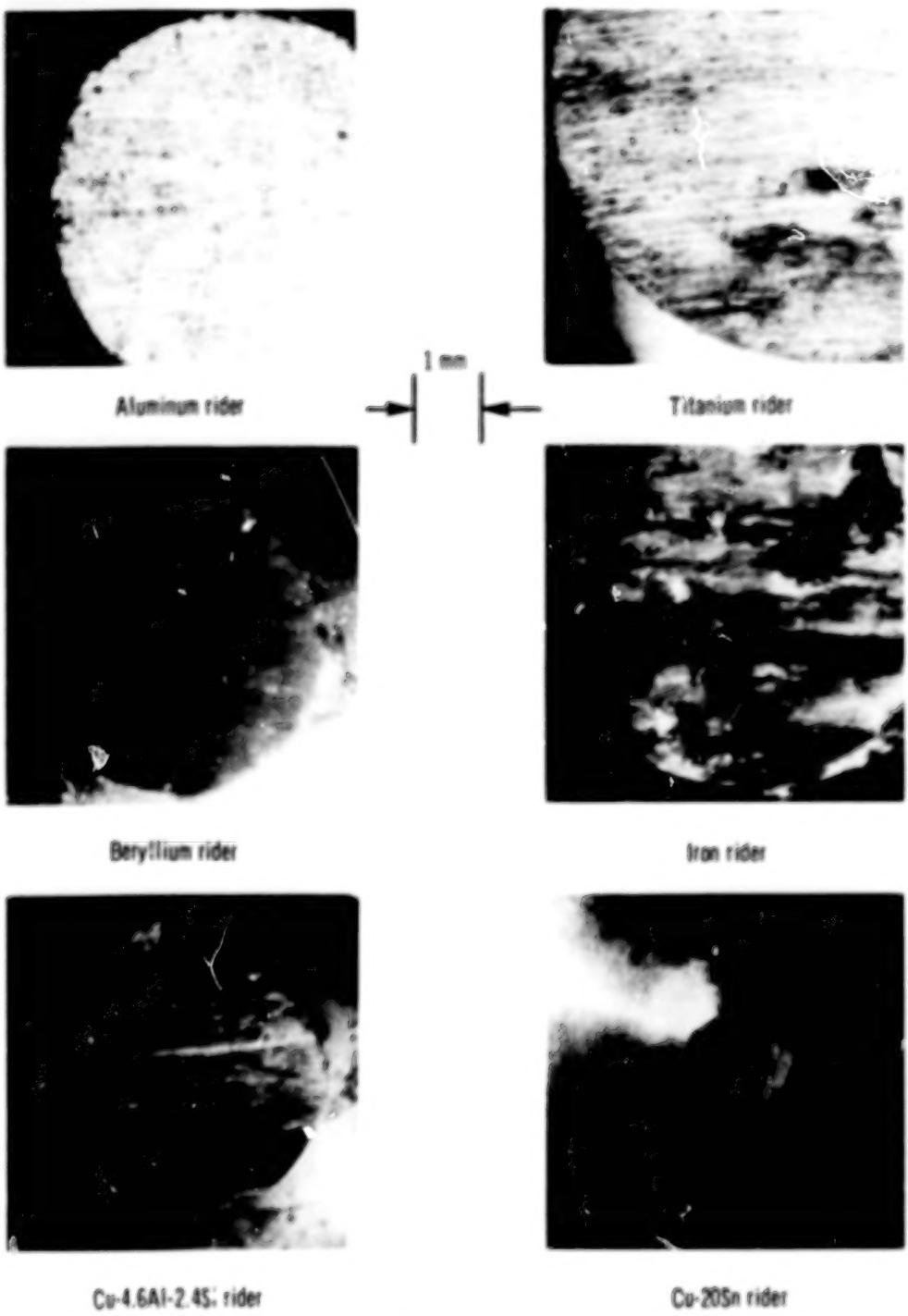


Figure 3. - Representative rider wear scars of metals run in sliding contact with 440C ( $R_c$  58). Load, 1 kilogram; sliding velocity, 12.4 meters per second; run submerged in liquid methane; duration of test, 30 minutes.

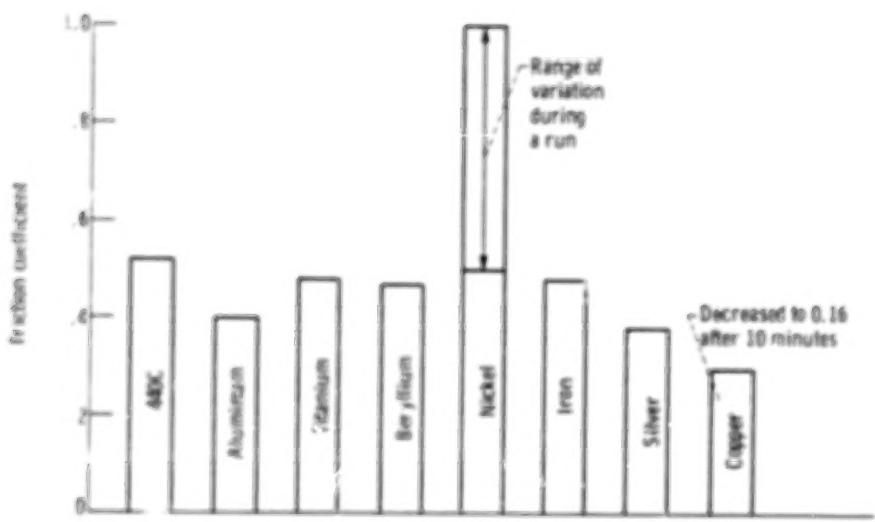


Figure 4. Friction coefficient of metal rods sliding against 480C in liquid methane. Load, 1 kilogram; sliding velocity, 12.4 meters per second (4200 rpm); duration, 30 minutes.

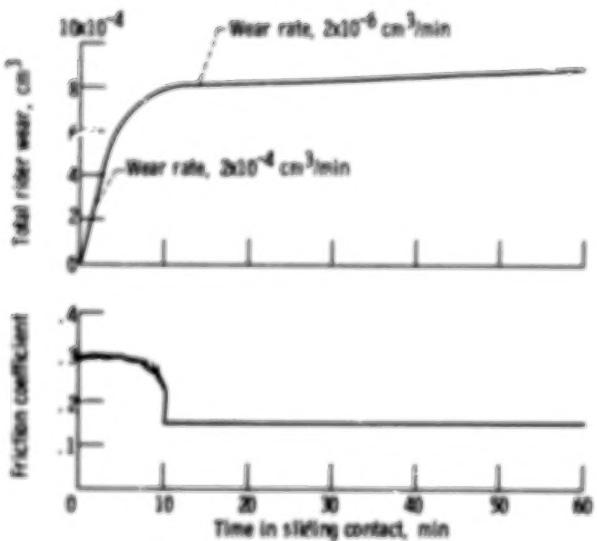
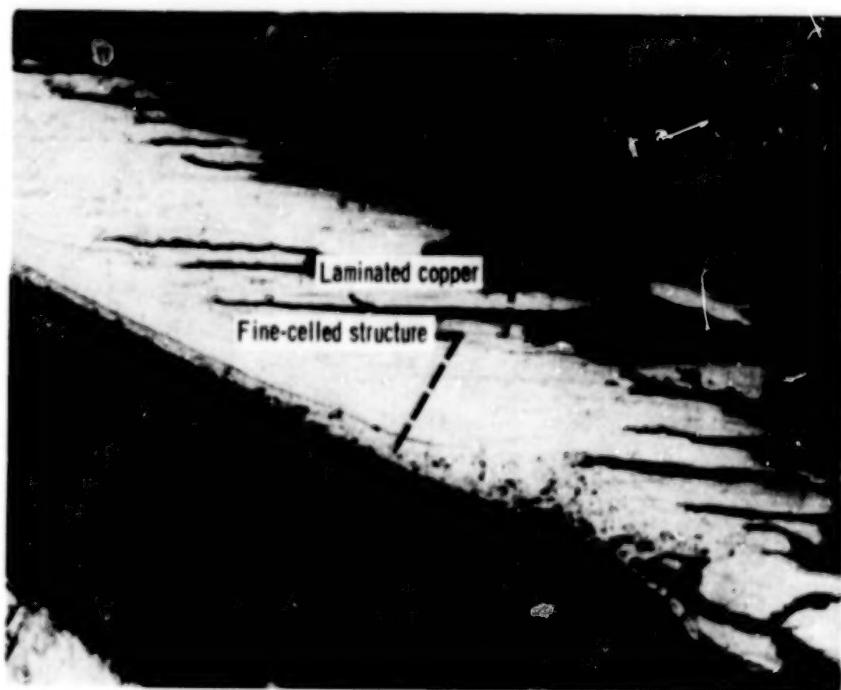


Figure 5. Typical friction- and wear-time trends for copper sliding against 480C in liquid methane. Load, 1 kilogram; sliding velocity, 12.4 meters per second (4200 rpm).



(a) Trailing wear debris (broken off).



(b) Very fine structured copper.

Figure 6. - Photomicrographs of copper rider trailing wear debris showing layers of copper and a very fine structured copper layer on lower surface of trailing wear debris ribbon. Load, 1 kilogram; sliding velocity, 12.4 meters per second; etched with picrol-HC1 250X.

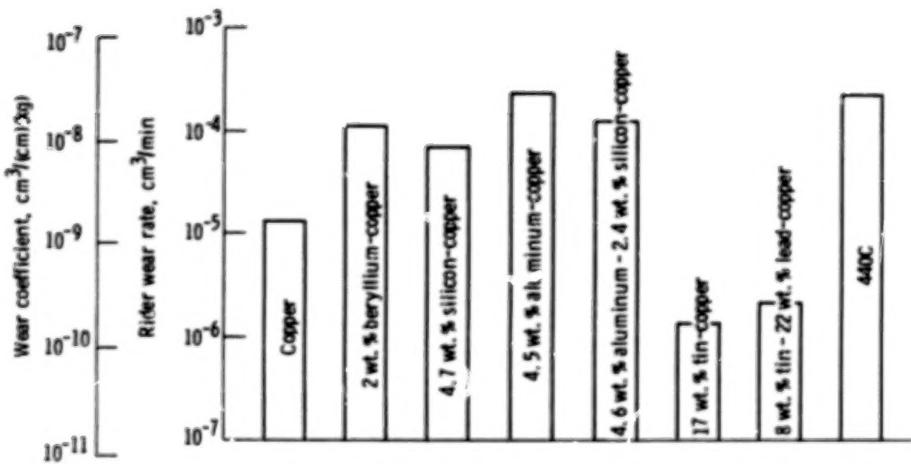


Figure 7. - Wear of copper and copper alloys sliding against 440C disks in liquid methane. Load, 1 kilogram; sliding velocity, 12.4 meters per second (4200 rpm); duration, 30 minutes.

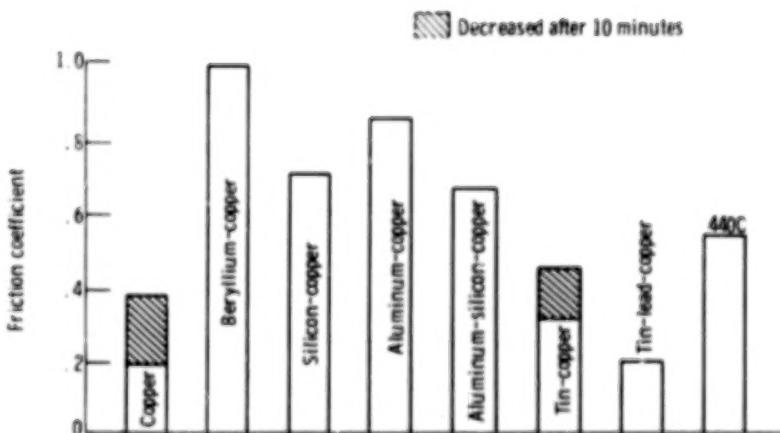
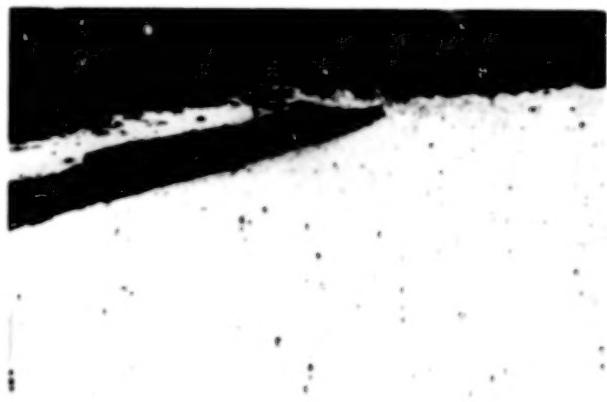


Figure 8. - Friction coefficient of copper and copper alloys sliding against 440C in liquid methane. Load, 1 kilogram; sliding velocity, 12.4 meters per second (4200 rpm); duration, 30 minutes.



Copper



Aluminum



Titanium



Iron

Figure 9. - Photomicrographs of selected metals run in sliding contact with AISI 440C stainless steel in liquid methane showing fine-grained structure at rub surface and severely cold worked layer directly underneath. Sliding velocity, 12.4 meters per second; load, 1 kilogram; direction of sliding, right to left. X250.

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16 Abstract <p>Aluminum, titanium, beryllium, nickel, iron, copper, and several copper alloys were run in sliding contact with AISI 440C in liquid methane and liquid natural gas. All of the metals run except copper and the copper alloys of tin and tin-lead showed severely galled wear scars. Friction coefficients varied from 0.2 to 1.0, the lowest being for copper, copper-17 wt. % tin, and copper-8 wt. % tin-22 wt. % lead. The wear rate for copper was two orders of magnitude lower than that of the other metals run. An additional order of magnitude of wear reduction was achieved by the addition of tin and/or lead to copper.</p>			
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